Interlaboratory Comparison Exercises for Organic Contaminants and Trace Elements in Marine Sediments and Tissues

Laboratories measuring contaminants in the marine environment must assess the accuracy and precision of their measurements. Quality control of measurements made on marine environmental samples is vital to the accurate assessment of marine pollution and its effects on wildlife and human health. Representative control materials needed to benchmark analytical methods and measurements are often limited in the number of compounds for which values are assigned, or are simply not available. Consequently, analysis may be undertaken on samples without control materials that are similar in nature to the sample. To help address this need, NIST, with the help of other U.S. Government sponsors, initiated several programs to assess the data quality of laboratories performing chemical measurements on marine-related samples.

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NIST helps benchmark and improve the quality of analytical data on the marine environment by conducting interlaboratory comparison exercise programs. The first such program for environmental measurements was initiated in 1987 and funded in part until 2000 by the National Oceanic and Atmospheric Administration's (NOAA's) National Status and Trends Marine Monitoring Program (NS&T). This program provides mechanisms for assessing the interlaboratory and temporal measurement comparability of polycyclic aromatic hydrocarbons, polychlorinated biphenyl (PCB) congeners, and chlorinated pesticides in bivalve mollusk, sediment, and fish samples. The program includes components for developing improved analytical methods, producing NIST Standard Reference Materials (SRMs) and other control materials, conducting annual interlaboratory comparison exercises, and coordinating workshops to discuss exercise results. This program continues as the NIST Intercomparison Program for Organic Contaminants in the Marine Environment with partial support from fees paid by the participants, and served as the model for the Interlaboratory Comparison Exercises Program for Organic Contaminants and Trace Elements in Marine Mammal Tissues.

NIST expanded the Interlaboratory Comparison Exercise for Organics in Marine Mammal Tissues in 1999, and the trace element complement to the exercise was formalized in 2000. Since this time, participation has increased dramatically. For example, the number of laboratories participating in the exercises has increased from 10 in 1999 to 20 in 2005. The exercises are international with nine countries involved in the trace element exercise and six in the organic exercise in 2005.

The organics exercise has been successful as a source of data on new constituents in SRM 1945. For example, fatty acids and brominated flame retardants were determined in the unknown and in SRM 1945 by a number of laboratories. The trace element exercise led to the development of three trace element control materials from marine mammal livers, a pilot whale liver homogenate in 1991, and beluga whale and pygmy sperm whale liver homogenates in 1997 and 2003, respectively. Independent analytical methods at NIST (inductively coupled plasma mass spectrometry and instrumental neutron activation analysis) produced data to provide a reliable benchmark to assess the performance of participating laboratories. The trace element exercise also used an innovative approach to arrive at a consensus mean and an associated uncertainty using a maximum likelihood solution model developed by Rukhin and Vangel at NIST.

Data from the monitoring program led to the development of a suite of control materials for trace elements in marine mammal tissues and the production of SRM 1945 Organics in Whale Blubber.

Impact: Comparability of measurements for organic contaminants has improved for those laboratories with a longer history of participation in the exercise, e.g., those that have participated since 1999 have improved their accuracy by 10% and their precision by 20%. The SRMs and control materials developed through this program and the interlaboratory comparison exercises are mandated for use by researchers in the US who are funded by NOAA to perform measurements of trace elements and organic contaminants in marine mammal tissues.

Future Plans: NIST will continue to administer and coordinate interlaboratory comparison exercises for chemical analysis of marine mammal tissues. Future exercises will request that participants provide measurements on compounds of emerging interest such as brominated and fluorinated compounds, toxaphene congeners, and organometallic contaminants, such as butyl tins and methylmercury. The next round of interlaboratory comparisons will occur in 2007. In 2006, the data compiled for SRM 1945 will be used to help update this material's Certificate of Analysis for compounds such as additional polychlorinated biphenyl congeners, brominated diphenyl ether congeners, toxaphene, and fatty acids.